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Technological aspects of high speed direct laser deposition based on heterophase powder metallurgy

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Abstract

The article deals with physical peculiarities and technology of high speed processes of direct laser deposition. On the base of theoretic research and computer modeling the powder transfer has been optimized, increasing process stability and productivity. Principles of nozzles design also have been developed in accordance with technological needs. An influence of process mode on product properties and material structure was defined for heat resisted Ni-based superalloys. Developed technology provided the mechanic properties of products on the level of rolled material and allows avoid heat treatment and HIP in production process. Possible ways for increasing process performance and economic efficiency also have been discussed.

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Keywords: high speed direct laser deposition, additive manufacturing, heterophase powder metallurgy, superalloys

1. Introduction

1. Introduction

Intensive development of additive technologies allows improves manufacturing methods for parts and products(Santos et al. 2006, Dutta et al. 2011, Murr et al. 2012, Turichin et al. 2015, Gu 2015,Kianiana et al. 2015).

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Additive technologies are replacing the conventional methods of casting and subsequent time-consuming machining which can remove up to 90% of costly material, increasing parts cost. The issue of cost reduction is especially relevant in high-tech industries (gas turbine engine, aviation, aerospace etc.) (Ageev et al. 2013, Wilson et al. 2014, Dorochov and Abacharev 2015).

Nowadays additive technologies based on selective laser sintering and selective laser melting (SLS- and SLM-technology) (Safin 2011, Olakanmi et al. 2015, SLM solutions 2015) almost ready for practical application. Despite the large number of studies conducted in this area, and availability of positive experience the potential of additive technologies is not fully implemented. The main development's trend of additive technology is increasing productivity while maintaining required quality of manufactured products.

The most perspective technology of products manufacturing is high-speed direct laser deposition (or Direct Metal Deposition, DMD), when the product is formed from a powder, which is supplied by compressed gas-powder jet directly into the laser action zone, wherein the jet can be as coaxial and as non-coaxial to focused laser beam. Laser provides heating and partial melting of the powder and heating the substrate (Turichin et al. 2015, Dutta et al. 2011 Wilson et al. 2014 Thompson et al. 2015). It is possible to use several materials and to modify the composition of powders directly during deposition, providing high-speed formation of products with gradient properties.

Research and development of deposition technologies require an integrated approach, i.e. the results of theoretical studies and mathematical modeling should be tested by experimental studies. Using this approach for development of new technologies and equipment allows increasing efficiency by decreasing of material and time costs and reducing the risk of negative outcomes. Results of theoretical and experimental investigations of laser cladding of metal layers and direct laser deposition from powder materials are presented in this paper.

2. Materials and research methodic

2.1. Processing conditions

Experimental studies of deposition processes were carried out at the Institute of Laser and Welding Technologies SPbPU (ILWT) using experimental set-up based on 5kW IPG fiber laser YLS-5000, 5D CNC machine, Sultzer Metco Twin 10-C powder feeder and HighYAG BIMO processing head (Figure 1).

Fig.1. Experimental set-up for direct metal deposition.

During experiments of DMD, following parameters were ranged: laser power, laser beam spot size, linear velocity, layer height, powder and transport gas feed rates, nozzle inclination angle, and nozzle standoff distance. Gas-powder jet studied separately at another experimental set-up, equipped with high-speed monochrome camera Citius Centurino C100, high resolution monochrome camera Basler acA-2000gm, lightening device based on 20W 808nm diode laser and laser line generator. Experiment automation was done in LabVIEW 2012 programming environment. Gas powder jets were formed by side and coaxial nozzles of different design. As a base material for direct laser deposition, powder alloy EuTroLoy16625G.04 Castolin Eutectic (Inconel 625) was used (fig.2), chemical composition is shown in Table 1. Fractional composition of 53-150 microns, the shape of the particles is spherical.

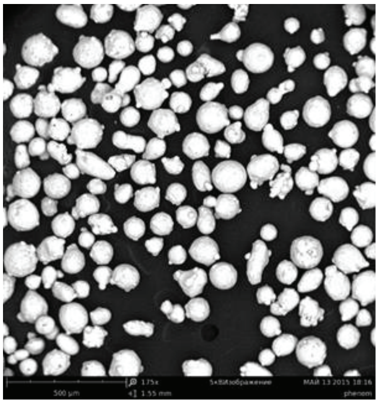


Fig.2. Granules of EuTroLoy16625G.04 alloy.

Table1. Chemical compound of alloy EuTroLoy16625G.04.

element	compound, %
Ni	base
C	0,00
Si	0,40
Nb+Ta	3,70
Cr	21,50
Mo	9,00
Fe	max. 5
Mn	0,35
Al	0,02
Ti	0,02
Others	0,10

Metallographic studies were carried out on microscope DMI 5000 (Leica) with Tixomet software. Researches of chemical composition and chemical elements distribution were made on scanning electron microscope Phenom ProX and Mira Tescan microscope using console Oxford INCA Wave 500. To determine the mechanical properties samples were tested on uniaxial tension, using universal testing machine Zwick/Roell Z250 Allround. Flat samples were tested in the initial state and in the state after heat treatment (annealing for releasing tension, T = 1000 ° C, 3 hours, air atmosphere). Heat treatment was made using furnace RHTV 120/300/1700, Nabertherm.

3. Experimental results

Development of direct metal deposition technology requires comprehensive theoretical and experimental research, careful selection of process parameters, a detailed study of the phase-structural state of produced layers and products, a comparative analysis of mechanical and exploitation characteristics also needed. Firstly, understanding of the gas dynamic and thermal processes in the gas-powder jet at the incidence on substrate whose value considerably exceeds the size of deposited bead is required. The next step is establishing of optimal cladding mode of single bead, which fit the requirements, and it's comprehensive analysis, including structural studies (Turichin et al. 2012). During the transition from single cladding layers to direct metal deposition, task of metal particles flight in the carrier gas jet is complicated by decreasing of specific surface area at which the growth product (previous layer). The theory of processes in the gas-powder jet when it falls on a substrate based on heating and partial melting of the powder particles under laser radiation has been developed by the authors of this study (Turichin et al. 2009). It include the joint solution of problems of incidence of the jet on the barrier and powder particles transfer in the jet. Mathematical model, based on developed theory of the powder transfer (Turichin et al. 2014), allowed connect the structure of gas-powder jet with flow rate of carrier gas, the nozzle size, and parameters of the powder particles. Experimental studies have also focused on the selection of process parameters to provide maximum utilization of the powder and providing of heterophase nature of the process with an incomplete melting of metal particles. It allows increases the mechanical properties of the product as compared to casting and SLS / SLM-technology.

Power feeding nozzle is an important part of technological head (Thompson et al. 2015, Leyens and Beyer 2015), which forms gas-powder jet and influences the deposition process. Researching processes of gas and powder flow through nozzles of different geometry - is key to understanding laser deposition. Nowadays deposition nozzles are commercially available from different companies. They have different design, different characteristics and price. But all of them can be classified into two big groups: coaxial and side nozzles. In the simplest case side nozzle - is a copper tube with inner diameter from 1 up to 3 mm, which is inclined to laser beam, pointed to focal point and located from 2 to 15 mm from active zone. Deposition material is carried with transport gas from powder feeder through nozzle to melt metal pool, partially or fully melts and forms a deposition bead. When technological head passes repeatedly several times deposited wall is formed. Thickness, height and roughness of this wall are a function of laser beam focal diameter, beam power, speed and powder jet geometry and density. Example of side nozzle is shown on picture 3 (left). Laser beam pass vertically through center part, which is water cooled and purged with argon to protect shielding glass. Metal powder is transported with 8 l/min of Argon through side copper nozzle with inner diameter of 2 mm. Inclination angle is 30°. Nozzle is situated 3mm from active zone. This set up allows produce walls from 0.5 up to 3 mm thickness, with rate up to 1 kg/hour. Linear speed is 40-45 mm/s, laser power 800-1000W, laser spot is 1.2 mm diameter, powder feeding rate is 20 g/min, and deposition level height is 0.2 mm.

Fig.3. Deposition head with side nozzle (left). Deposited samples (middle and right).

Side nozzles have simple geometry and easily can be modified, that means that deposition process can be studied in detail. By changing nozzle inclination angle, inner diameter, spot size and nozzle standoff different set-ups can be studies. A number of experiments were done in order to find out correlations between these technological parameters and wall characteristics. For example, it is possible to control deposited wall thickness by adjusting active zone size through controlling of laser power and spot size. Automatic control system based on analysis of video image of melt pool, captured by coaxial camera, can adjust wall thickness in real time.

One of most important characteristic of experimental set-up is process robustness. During deposition previous layer can be situated lower or upper than predicted, due to process instabilities, errors of head movement, thermal deformations and other problems. Robust process should neutralize these instabilities without external intervention. If previous layer somehow is located lower some optimal level powder jet geometry should cause deposition of higher bead. If previous layer if located upper, deposited bead should be lower. Simple design of side nozzles gives more capabilities for these experiments.

Side nozzles have one important disadvantage - they are not symmetric referring to laser beam. That means that deposited bead geometry depends on movement direction. This limits variety of shapes, which can be achieved to revolution bodies. For deposition of free-shape items coaxial nozzles are used - they are laser beam symmetric and isotropic to movement direction. These nozzles are commercially available at two designs: with multiple jets and annular slit, two of them shown on figure 4 (Ocyloka et al. 2014, Frauhofer 2015).

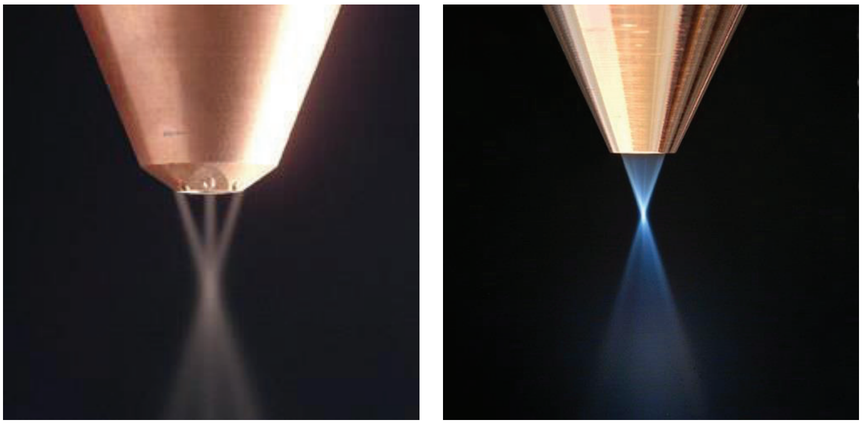


Fig.4. Coaxial nozzles with multiple jet (left) and annular slit (right) designs.

Multiple jet nozzle have from 3 up to 6 separate channels, which forms powder jet. Second design is more complex, but allows more flexibility for powder jet control - by adjusting slit width geometry and density of powder jet can be precisely controlled, that why it was chosen for experimental research. In order to figure out optimal parameters of nozzle a series of experiments were carried out.

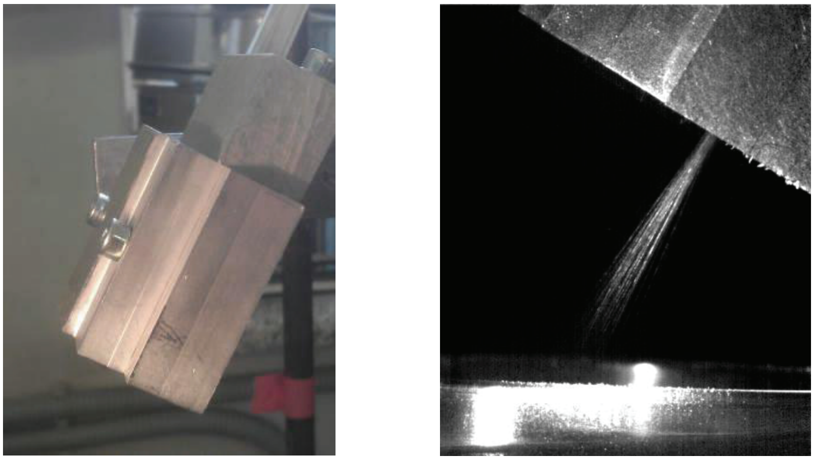


Fig.5. Overview of model experiment with flat slit nozzle (left), captured powder jet geometry (right).

Experimental set-up, shown on figure 5, was used to find dependence between slit width and geometry of powder jet. Flat nozzle was used as a simplified model of annual slit nozzle. Powder jet was back lighted with diode laser line generator and captured on Basler acA-2000-50gm digital camera. A sequence on 500 frames were captured during each experiment, frames were summed to get statistically proved distribution. Following range of parameters was used: transport gas (Argon) flow - from 3 up to 10 l/min, powder mass feed rate - from 5 up to 20 g/min (312L stainless steel powder with fraction 50-150 um), slit width - from 200 up to 1000 um. Three examples of captured and processed images are shown on figure 6.

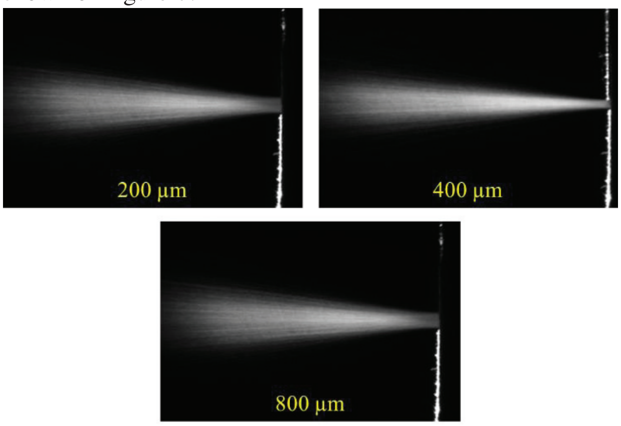


Fig.6. Powder jet after flat slit nozzle with 200, 400 and 800 μm gap.

It was carried out that gas flow rate slightly influence jet distribution as well as powder mass flow rate. The main factor affecting the width of the gas-powder jet after exiting the nozzle, is a slit width. Figure 7 shows measured jet width at distances of 5, 7.5 and 10 mm from nozzle exit.

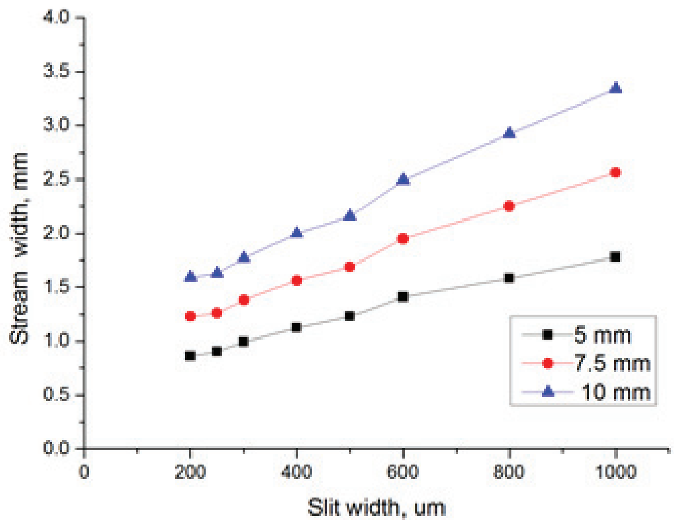


Fig.7. Dependence of jet width from slit size.

It is shown that there is linear dependence between these parameters, so geometry of powder jet can be precisely controlled. Figure 8 shows dependence of jet divergence angle from slit width.

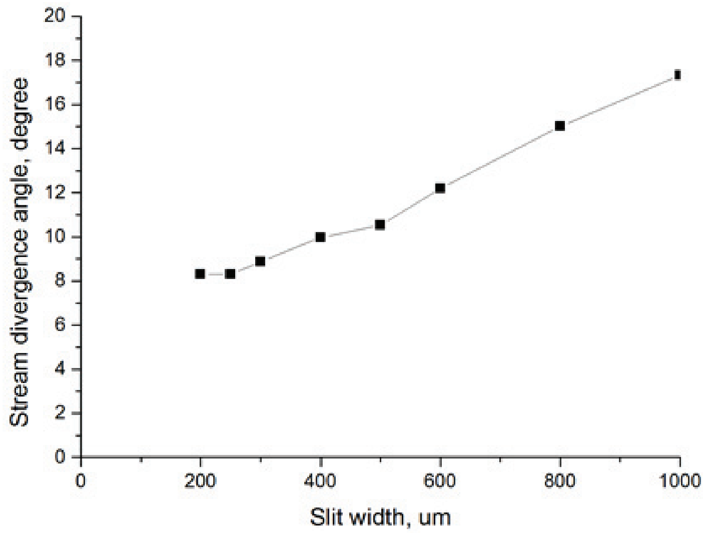


Fig.8. Dependence of jet divergence angle from slit size.

Divergence angle also increases monotonically with slit width increasing. Jet density distribution is close to normal and slightly affected by changing of chosen parameters. For further research annular slit nozzle with slit width of 0.3 mm, convergence angle of 60° and standoff distance 7.5 mm was chosen as optimal.

Another problem of annular slit nozzles - uneven powder distribution around the circumference was solved with two approaches. First step - gas-powder mixture, supplied from powder feeder was divided into 4 even jets by means of specially designed jet splitter with imprecision of 0.5% maximum. The second step was special annular cavity at upper part of nozzle gas-powder mixture is injected in this cavity in four points, twists around and then fed into annular slit though flow straightener. Final nozzle design is shown on figure 9.

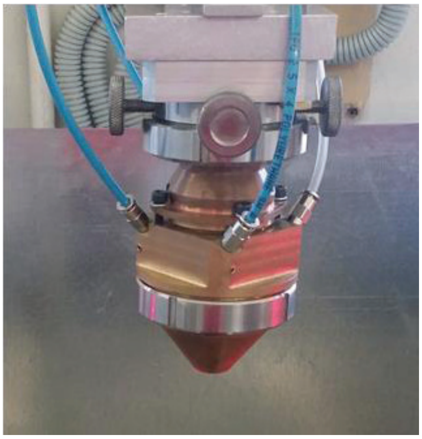


Fig.9. Coaxial nozzle with adjustable annular slit.

Samples with rotation body were made from superalloy Inconel 625 using selected technological parameters. Cross section of sample is represented on fig.10.

a) b)

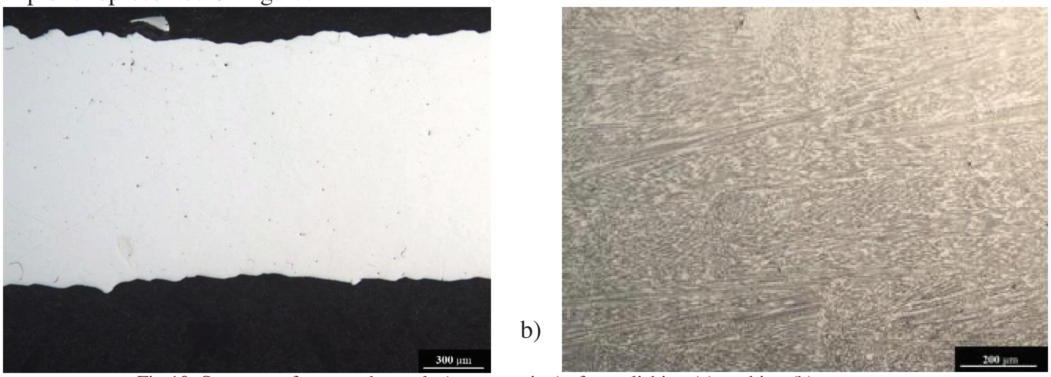


Fig.10. Structure of prepared sample (cross-section) after polishing (a), etching (b).

Porosity of samples less than 0.05 vol.%, there are no cracking and non-metallic inclusions. That proves, it's possible to made samples with good quality and protect from oxidation with local gas shielding. The microstructure mostly has cast condition; the longitudinal size of the dendrites varies in the range of 50-250 microns, the size of individual dendrites up to 500 microns. Layered structure with abrupt boundaries between deposited layers, which can be seen in the products manufactured using the standard SLM technology (Li et al. 2015), was not detected. That's also differ developed HSDLD process from simple DMD technology. Cast structure formation could also be related to the initial powder structure, which is produced by gas atomization. On fig.11 microstructure of used powder (a) and manufactured sample with power of 0,5kW (b) are shown.

a) b)

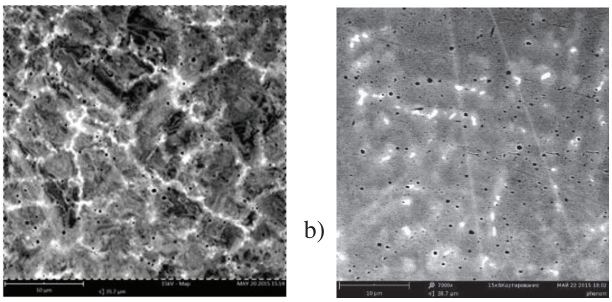


Fig.11. Microstructure of used powder (a) and manufactured sample (b).

Based on the results of spectral microprobe analysis and mapping of the selected area, the gray region is nickel-based gamma solid solution, white grid along the grain boundaries - niobium and molybdenum carbides, black dots is a finely dispersed silica, alumina and manganese oxide (Shamsaei et al. 2015, Reed 2006). Produced samples have inherited microstructure of using powder. During HSDLD-process dissolution of carbide grid and subsequent isolation to the separate inclusions with dispersion 0.5-2 μm are occurred, size and form of oxides during direct laser deposition is unaffected.

To evaluate the effect of microstructure of manufactured samples to the mechanical properties heat treatment was carried out - (annealing to release tension, T = 1000 ° C, 3 hours). The results of the tensile tests are shown in Figure 12.

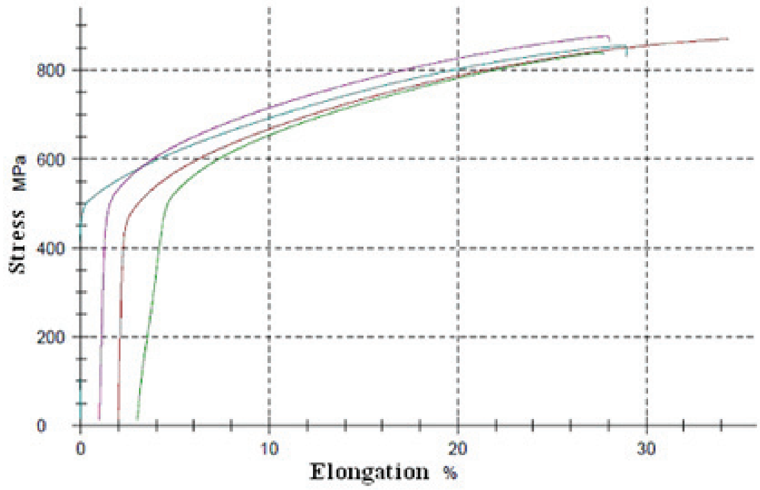


Fig.12. Tensile test curves for specimens from alloy EuTroLoy 16625G.04.

Mechanical properties of the alloy before the heat treatment: tensile strength on average is 866 MPa, the yield strength - 488 MPa, elongation - 28%. The mechanical properties after heat treatment: tensile strength on average is 855 MPa, the yield strength - 479 MPa, elongation - 27%. These values match to the reference data of the properties of INCONEL alloy 625 in rolled condition: tensile strength - 827-1103 MPa, yield strength - 414-758 MPa, elongation - 60-30%. To evaluate the type of destruction the fractographic study was made, the results are shown in Figure 13.

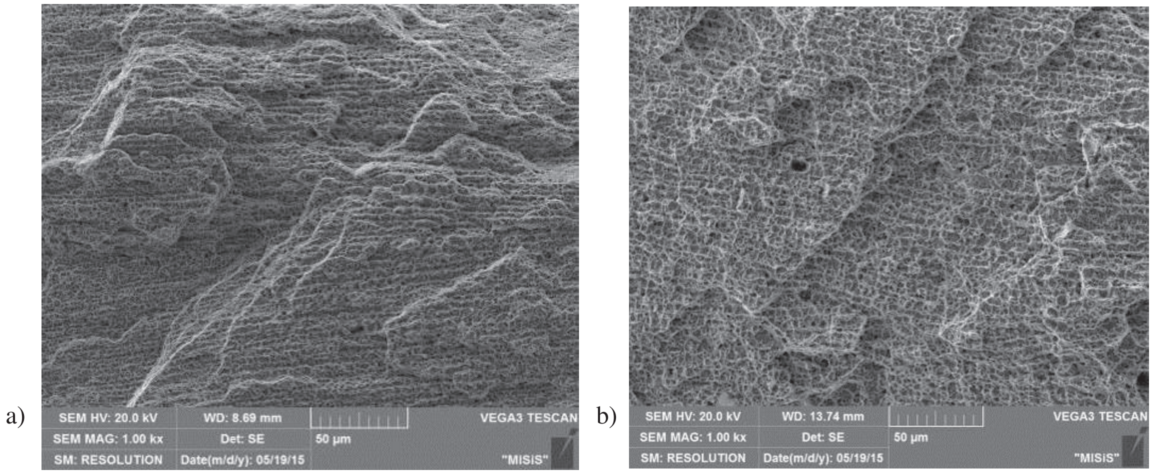
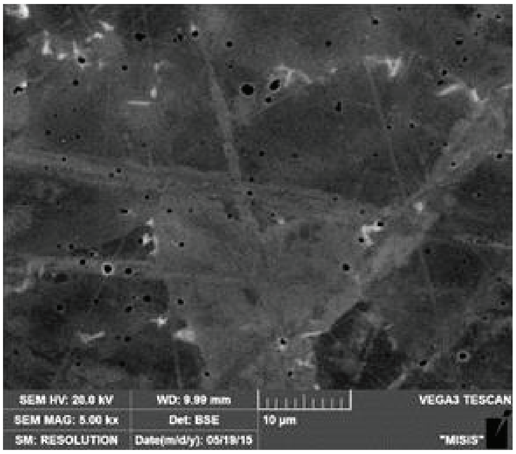


Fig.13. Fractures structures of samples after mechanical uniaxial tensile tests before (a) and after (b) heat treatment.

The photographs show large amount of facets with traces of plastic deformation, which indicated viscous nature of the fracture. It should be noted fine grained facets in samples without heat treatment are occurred. Samples after heat treatment also have a large number of facets with traces of plastic deformation, which also show the viscous nature of the fracture.

Comparing the microstructure of polished and etched samples (Figure 14) before and after heat treatment, should be noted, that a fine grain structure in both cases are occurred.

a) 

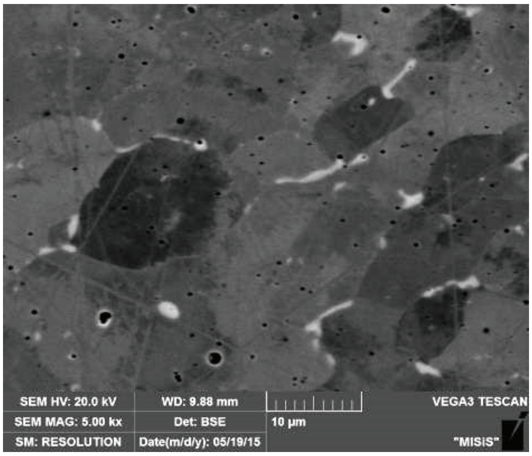
b) 

Fig.14. Microstructure of samples from EuTroLoy 16625G.04 alloy in initial (a) and heat treated condition (b).

The structure contains regions with small and large grains, which indicates certain unevenness. After the heat treatment the size of carbide and oxide inclusions does not change as the size of the grains. Mechanical properties are remaining at the level of rolled condition, so it could be argued that the process of direct laser deposition allows manufacturing of products with satisfactory performance. Further heat treatment is not required.

4. Conclusions

Direct laser deposition technology is complex and multifactorial process with a lot of parameters, which affect to the final result. Therefore, to understand the relationships between process parameters and optimize the technology of products with specified characteristics, also for reducing material and time costs and risks of negative results, the study should be carried out comprehensively. So, theoretical research and mathematical modeling should be accomplished by experimental investigation, proving results one for another.

Research set-up for experimental studies of gas-dynamic processes of powder transfer allowed studying the effect of nozzle geometry, transport gas flow and fractional composition of the powder on spatial structure of the gas-powder jet. The results of experimental studies of gas-dynamic processes are in good agreement with results of mathematical modeling, which confirms the physical adequacy of developed mathematical model. The following process parameters are recommended: the width of the gap of 250-300 mm, the angle of convergence of the nozzle cone of 60°, the distance from the nozzle to the area of the powder jet waist is 9-10 mm. The mechanical properties of the alloy EuTroLoy 16625G.04 before and after heat treatment are at the same level as the rolled metal: tensile strength averages 850-900 MPa, yield strength - 470-490 MPa, elongation – 28-30%. The structure of the samples is characterized by equiaxed grains with a second release phase (mainly carbide) at the grain boundaries. The microstructure is inherited from the used metal powder. The fracture structure has a facets character, which peculiar to materials with a sufficiently high ductility.

The research results showed, that developed technology of direct laser deposition, can replace the currently used technologies, providing multiple increase productivity and material savings, in spite of its technological complexity. Products made by direct laser deposition without further isostatic pressing, or heat treatment, require significantly shorter time of manufacturing in comparison with SLS / SLM - technologies and technologies, based on casting and subsequent heat treatment and machining.

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